

Quarterly Progress Report  
For  
Mercury Removal in a Non-Thermal, Plasma-Based  
Multi-Pollutant Control Technology for Utility Boilers

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This report describes progress Powerspan made from October through December, 2002 on specific tasks listed in the Cooperative Agreement. Powerspan worked on and charged costs to the following tasks:

- Task 1: Install and Test Mercury Monitoring Equipment
- Task 2: Baseline Measurements
- Task 4: Determine the Effect of Wet Electrostatic Precipitator and Scrubber Operating Parameters on Mercury Removal
- Task 6: Determine Processes to Remove Mercury from Liquid Effluent Streams
- Task 7: Technology Transfer

Reports on the status for each of these tasks as well as other relevant data are presented below. During this reporting period the ECO Pilot flue gas supply was switched from the R.E. Burger Plant's Unit 4 to Unit 5. The switch was done to allow the pilot to draw flue gas from a boiler with low NO<sub>x</sub> burners and to allow for continued pilot operations during a planned shutdown of Unit 4. The differences in flue gas between Units 4 and 5 are expected to be only those due to the low NO<sub>x</sub> burners on Unit 5. The two Units are of identical boiler design and are supplied from the same fuel source.

A major focus of the work performed by Powerspan during this quarter has been measurement of elemental mercury (Hg<sup>0</sup>) in the inlet flue gas stream. The problem of accurately measuring elemental Hg is one that has implications for control technologies throughout the coal-fired Utility industry. Ash present in the gas stream can oxidize Hg, particularly on filters and other equipment used to condition gas samples for Hg measurement, biasing the measurement towards oxidized Hg (Hg<sup>2+</sup>). Control of Hg<sup>0</sup> emissions is known to be more difficult than controlling Hg<sup>2+</sup> emissions and processes implemented for Hg<sup>2+</sup> control are not certain to be reliable for Hg<sup>0</sup> control. As a result techniques for accurate measurement of Hg<sup>0</sup> are essential for development and application of Hg control technologies

### Task 1: Install and Test Hg Monitoring Equipment

Accurate measurement of mercury concentrations in the flue gas stream entering the ECO<sup>®</sup> Pilot has been problematic. The previous Quarterly Progress Report (November, 2002) stated that installation and testing of mercury measurement equipment was essentially complete. However, subsequent testing with elemental mercury ( $\text{Hg}^0$ ) addition to the flue gas stream has shown that measurement of  $\text{Hg}^0$  is not accurate with the sample conditioning equipment supplied by PS Analytical (PSA). Ash collected on the filter in the conditioning system reacts with  $\text{Hg}^0$  in the gas, oxidizing it to  $\text{Hg}^{2+}$ . As a key aspect of successful demonstration of the ECO process for Hg removal is oxidation and capture of  $\text{Hg}^0$ , an accurate measurement of  $\text{Hg}^0$  in the inlet flue gas is essential. Tasks 2 through 5 cannot be completed until valid measurements of  $\text{Hg}^0$  are available.

There have also been several issues with Hg measurement equipment reliability in addition to the problem of measurement bias towards  $\text{Hg}^{2+}$  in the presence of reactive ash. The major difficulties with the installed PSA semi-continuous Hg systems identified to date are listed below:

1. Oxidation of  $\text{Hg}^0$  to  $\text{Hg}^{2+}$  that occurs on the conditioning system ash filter in the presence of reactive ash, resulting in a significant measurement bias towards  $\text{Hg}^{2+}$
2. The instrumentation as provided has no built in ability to calibrate the entire system (probe, conditioning system and analyzer) using known quantities of Hg
3. Insufficient ash removal in the sample conditioning system, allowing Hg contaminated ash to reach the sample impingers and report as gas phase Hg
4. Unreliable operation of system components leading to:
  - a. Formation of precipitates in the impingers
  - b. Inadequate moisture removal
  - c. Unstable sample line flow control
  - d. Gold trap contamination

Several modifications to the PSA Hg measurement equipment have been made to improve system reliability. Impinger solution chemistry has been altered to prevent precipitation and to scrub acid gases in order to limit gold contamination. Excess sample flowrate has been reduced to provide better sample flow control and also to improve moisture removal from the sample gas.

The problem of ash contamination in the gas phase measurements can be seen in the total mercury concentration values recorded with clean (blank) gas fed to a PSA supplied probe. Figure 1 presents results from blank measurements made with the probe after using the probe to sample inlet flue gas for several hours. While sampling the probe's filter was blown back with compressed air after each sample in an effort to remove accumulated ash.

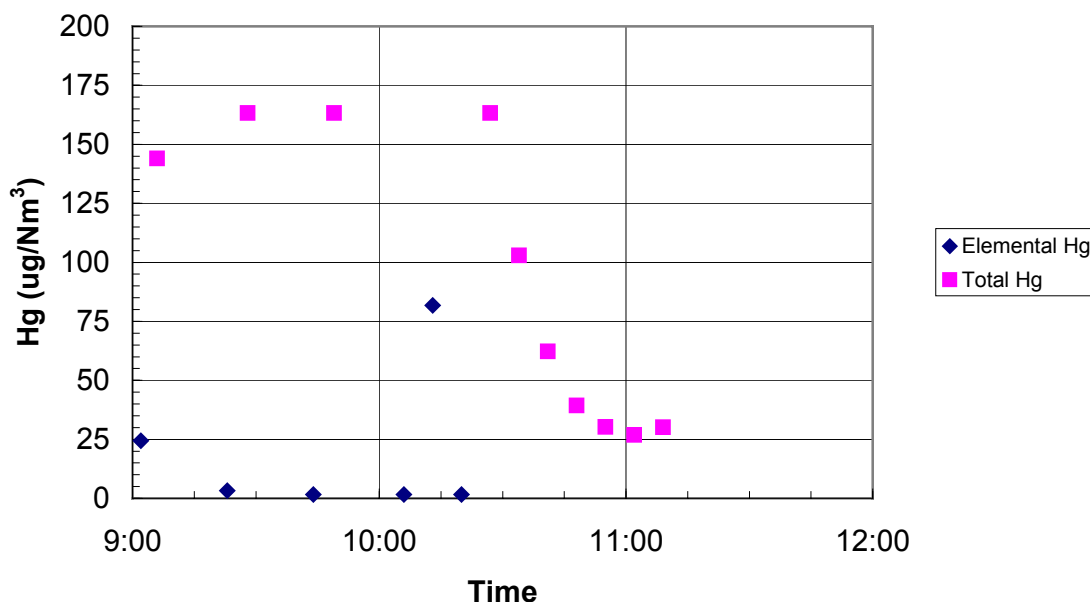


Figure 1 - Blank Hg Measurements with Ash Laden Probe

The exceedingly high ( $>100 \mu\text{g}/\text{Nm}^3$ ) blank values shown in the figure, along with the fact that the values decrease over time are evidence of severe contamination in the sampling system. As a check the system used to acquire the data shown in Figure 1 was cleaned and the blank measurements repeated. The process included cleaning of the sample lines and sample pump head with a 10% nitric acid solution followed by a rinse

with de-ionized water. Figure 2 presents the results of blank measurements made with the cleaned probe.

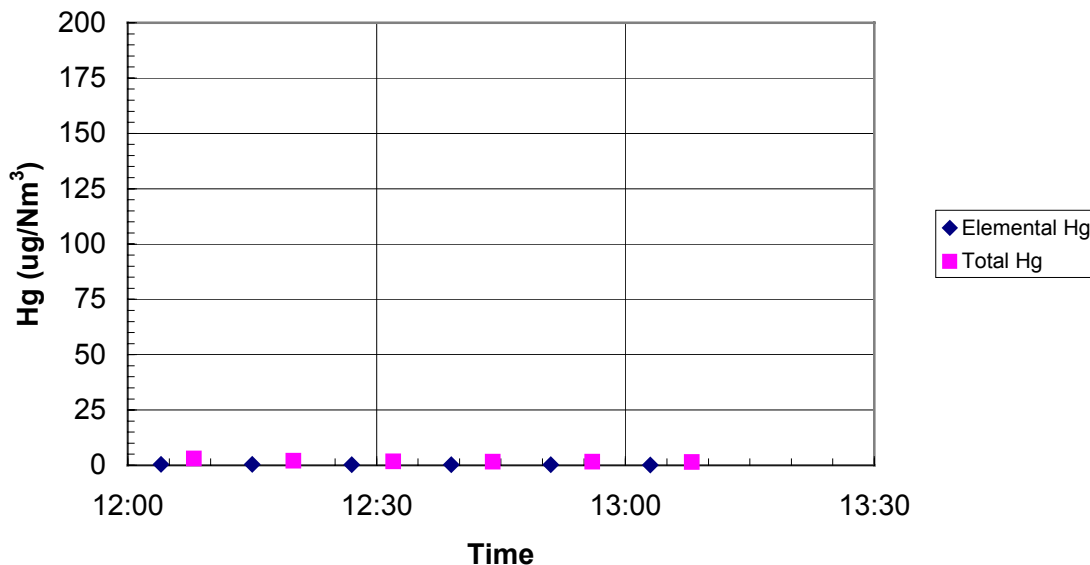


Figure 2 - Blank Hg Measurements with Clean Probe

Contamination of the sample lines and pump head with ash as evident from the data in Figures 1 and 2 was found to be due to deformation of the Teflon filters supplied with the PSA probes. Deformed filters allowed gaps between the filter and housing and for ash to enter the sample lines. Eventually the ash entered the impingers, where Hg contained on the ash was digested and released, reporting as gas phase Hg. When measured by the PSA instruments, it is seen as large spikes in the total Hg measurement. Ceramic and sintered metal filters were installed and tested in order to solve the problem of filter deformation. Several filter blowback configurations were also tested during this period to determine the most effective means of removing ash after each sample. However, during this testing a determination was made that simple filtering of the flue gas to remove particulate matter, including blowback cleaning of the filter after each sample, would not provide accurate speciation of mercury in the inlet gas stream. This is discussed further in the following text.

Flue gas provided to the ECO Pilot from FirstEnergy's Units 4 and 5 contains very little elemental Hg due to the fuel used at the R.E. Burger Plant and the boiler characteristics.

Therefore, an elemental Hg injection system was designed and installed so that the ability of the ECO reactor to oxidize  $\text{Hg}^0$  could be demonstrated. Initial testing of the  $\text{Hg}^0$  injection system showed that the majority of Hg introduced to the flue gas reported as  $\text{Hg}^{2+}$  instead of  $\text{Hg}^0$ . Figure 3 shows Hg measurements made at the inlet to the ECO pilot with and without Hg addition using the PSA supplied probe and a ceramic filter.

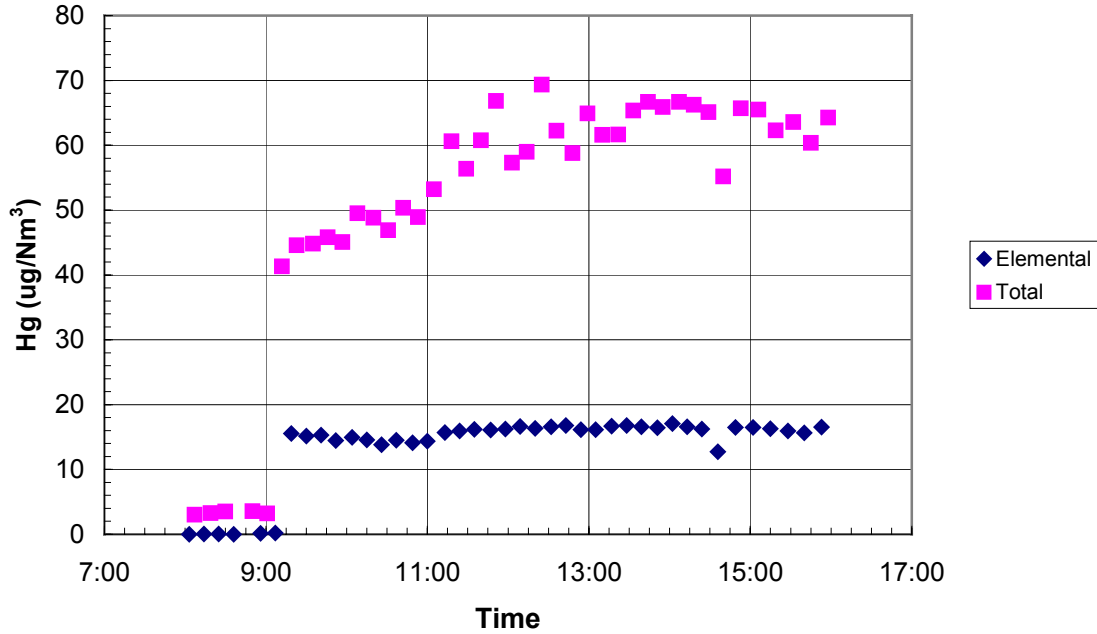


Figure 3 - Inlet Hg Concentration with Hg Addition

Flow from the Hg addition system was initiated at 9:10, at which point the total Hg concentration, as seen in Figure 3, increased from approximately  $4 \mu\text{g}/\text{Nm}^3$  to over  $40 \mu\text{g}/\text{Nm}^3$ . The Hg total concentration continued to rise over the next 5 hours until a value of approximately  $65 \mu\text{g}/\text{Nm}^3$  was reached. The concentration of  $\text{Hg}^0$  increased from  $<1 \mu\text{g}/\text{Nm}^3$  to approximately  $16 \mu\text{g}/\text{Nm}^3$  as a result of the Hg addition. The small increase in  $\text{Hg}^0$  ( $\sim 16 \mu\text{g}/\text{Nm}^3$ ) and large increase in total Hg ( $\sim 61 \mu\text{g}/\text{Nm}^3$ ) corresponds to an increase in reported  $\text{Hg}^{2+}$  of  $45 \mu\text{g}/\text{Nm}^3$  or 74% of the added Hg. Although reactive ash is clearly oxidizing a large portion of the added elemental Hg, the spikes seen with the Teflon filters indicating ash contamination are not seen with the ceramic filter. Repeated testing of the Hg addition system showed that the majority of injected Hg was measured as  $\text{Hg}^{2+}$ . In addition, material balance calculations on the Hg addition process indicated

that a significant portion of the injected Hg was captured by ash on the sample filter and did not report as gas phase Hg. The composition and amount of ash in the inlet flue gas stream is responsible for the oxidation of injected Hg. To characterize the ash loading on the ECO system measurements of the ash concentration and particle size distribution at the inlet to the ECO reactor were made by Air Compliance Testing (ACT) in November using EPA test methods. Table 1 presents the results obtained using EPA Method 5 sampling at two flue gas flow rates.

	Gas Flow (acfm)	Average Ash Loading (grains/dscf)	Range of Ash Loading (grains/dscf)
High Flow Conditions	3660	0.137	0.117 to 0.163
Low Flow Conditions	2920	0.130	0.089 to 0.153

Table 1: ACT Ash Loading Measurement Results

Presented in Table 2 is the result of particle size distribution measurements made by ACT.

Particle Size ( $\mu\text{m}$ )	High Flow Conditions (% Particles Below Size)	Low Flow Conditions (% Particles Below Size)
10.40	13.02	47.09
7.60	11.76	39.50
5.20	8.94	27.83
3.50	6.31	19.09
2.20	4.74	12.16
1.08	2.60	7.08
0.68	1.35	5.95
0.46	0.53	5.01
0.00	0	0

Table 2: Particle Size Distribution Measurement Results

The ash loading on the ECO pilot (0.14 to 0.15 gr/dscf) is over 10 times that measured at the outlet of the Unit 5 ESP (0.009 gr/dscf). In order to provide flue gas with an ash loading representative of that measured at the outlet of the Unit 5 ESP Powerspan is investigating the installation of a baghouse at the inlet to the ECO system. As an



alternative, installing ductwork to allow the ECO pilot to draw flue gas from the Unit 5 ESP outlet is also being considered.

Powerspan consulted with Sharon Sjostrum of EMC Engineering in November at the ECO pilot to determine methods to improve reliability of the Hg sampling equipment and to reduce the influence of ash on the Hg measurements. A Quicksilver Inertial Separation (QIS) probe from Apogee Scientific was purchased and installed on the flue gas inlet duct in December. The QIS probe uses inertial separation and filtration to extract a flue gas sample with little interaction between the gas and ash contained in the gas. Initial results with the probe are shown in Figure 4.

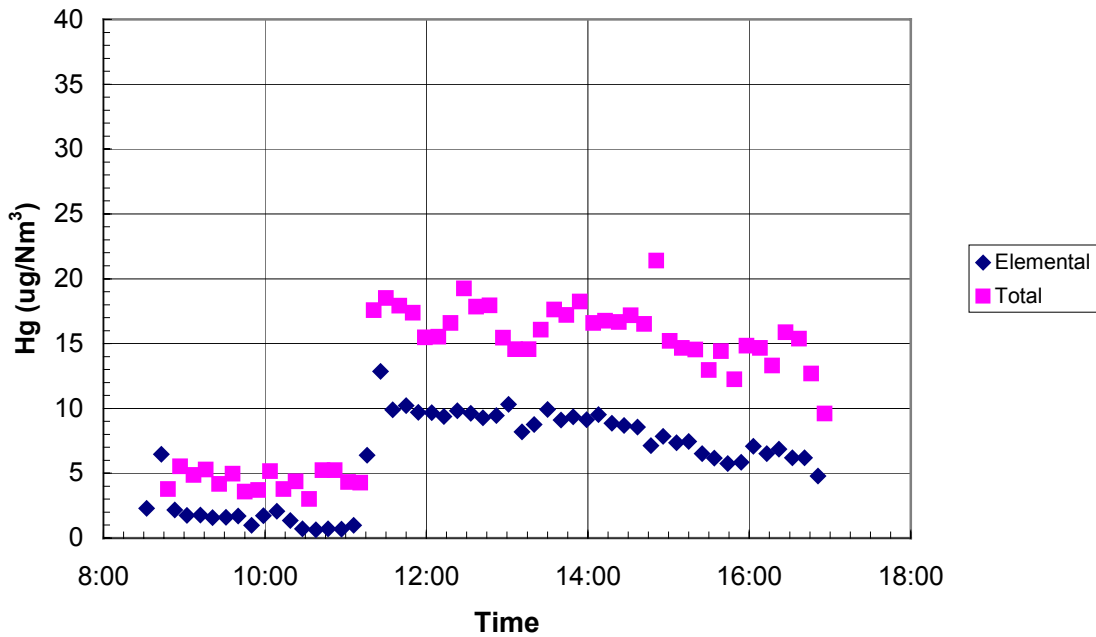


Figure 4 - Hg Measured with QIS Probe and Hg Addition

The data presented in Figure 4 shows that approximately 67% of the injected mercury is measured as  $\text{Hg}^0$  using the QIS probe, compared to 26% when the PSA supplied probe was used.

The ability to speciate Hg in flue gas containing reactive ash, such as at the inlet of the ECO reactor, is substantially improved with the QIS probe. At the outlet of the ECO scrubber and at the system outlet ash loading is low due to collection in the scrubber and

further collection in the Wet ESP. PSA supplied probes using a ceramic filters are considered to be acceptable for Hg species measurement in these locations.

Calibration checks of the entire sample system, from the probe to the analyzer are required to ensure the reliability of the Hg measurement. The PSA instrumentation does not provide for this capability. The atomic fluorescence analyzer is calibrated routinely. The calibration is done directly to the analyzer and bypasses the sample train. For system checks, a CAVkit supplies only an approximate amount of elemental mercury to the instrument through the probe. Figure 5 shows the results of several blanks and several samples drawn with  $\text{Hg}^0$  supplied to a PSA supplied probe with a clean ceramic filter. As expected, the blank values are low. The difference seen in the figure between the measured total Hg and  $\text{Hg}^0$  has been explained by PSA as due to a variation in the CAVkit supply of  $\text{Hg}^0$  to the probe when sampling for  $\text{Hg}^0$  and total Hg. The results presented in the figure point out the need for the ability to deliver a known concentration of Hg to the probe in order to perform probe calibrations.

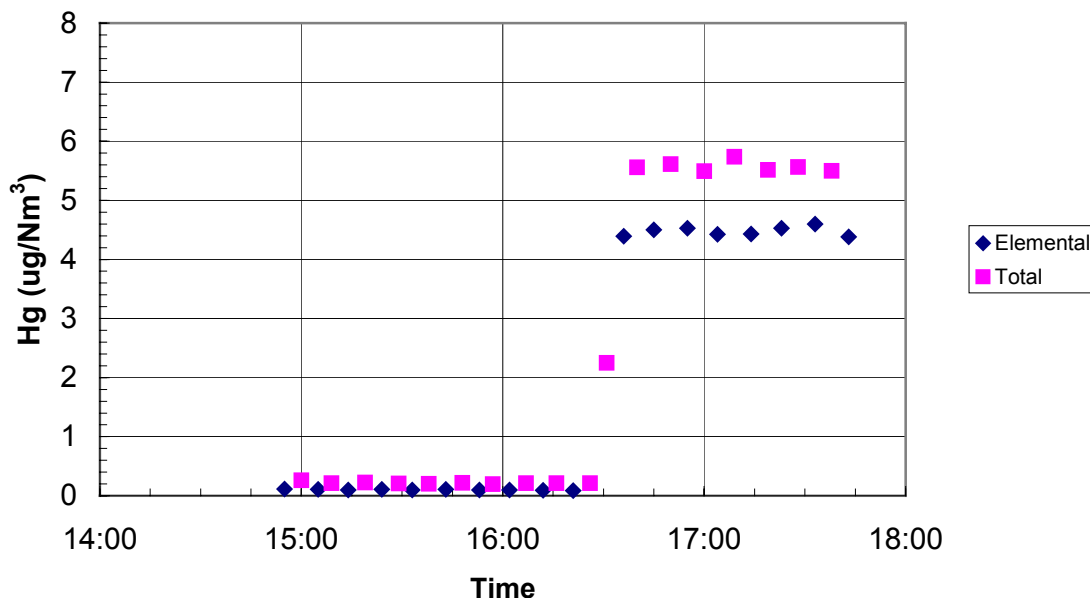


Figure 5 - Blank and CAVkit Results on Clean Ceramic Filter

Efforts have begun to provide for checks of the entire sample train. The checks will include installation of a batch sample train on each probe and injection of a known

quantity of oxidized and elemental Hg into the batch train. Measurements made with the batch sampling system will then be compared to those made with the semi-continuous systems. The batch sampling system has been constructed and tested in Powerspan's laboratory and will be installed at the ECO pilot in February.

## Task 2: Obtain Baseline Measurements

During this quarter, several days of data measuring Hg concentration at the inlet and outlet of the dry ESP using a single PSA instrument was obtained. The instrument was stream switched between two PSA probes with Teflon filters. Filter cleaning with blowback air was performed after each sample. Figure 6 presents the total Hg measurements recorded over approximately 32 hours of sampling. The data shows several samples with high ( $>40 \mu\text{g}/\text{Nm}^3$ ) concentrations indicative of ash contamination as discussed earlier in this report. Elemental Hg measurements were also made but were considered to be unreliable and are not presented due to the problems of reaction with ash previously discussed.

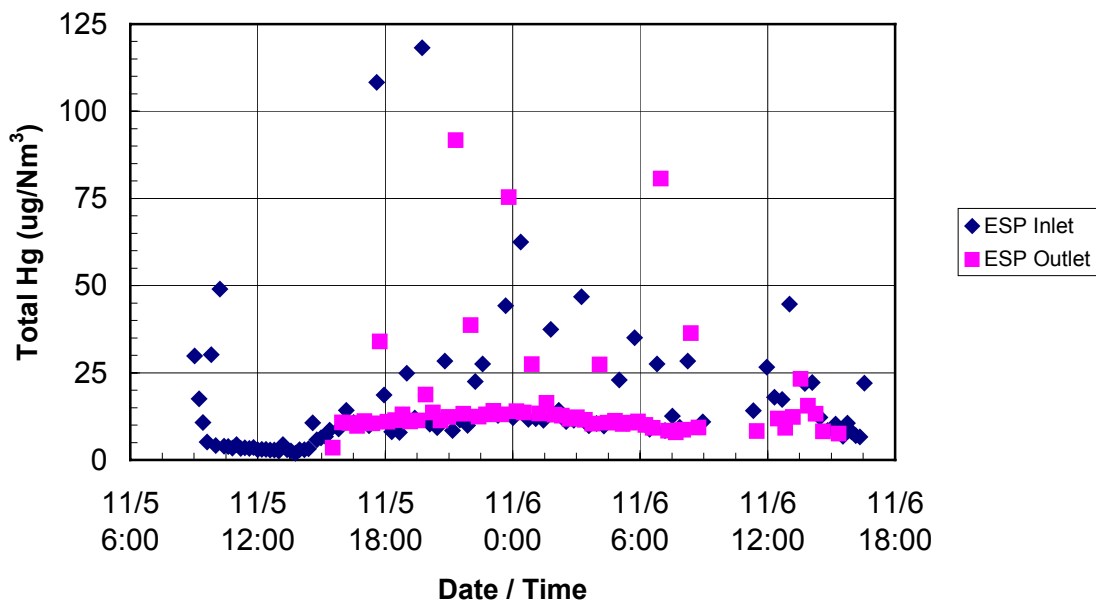


Figure 6 - Hg Measurements Across the Dry ESP

The Hg addition system was put into operation at 14:30 on 11/5. Measurements made prior to the addition of Hg show the total inlet Hg is less than  $5 \mu\text{g}/\text{Nm}^3$ . The values

recorded without Hg addition are considered to be too low for adequate testing of the ECO process and demonstration of its ability to oxidize and capture Hg. Additional baseline testing will be conducted with Hg<sup>0</sup> addition to determine operating conditions that will provide 10 to 40 µg/Nm<sup>3</sup> at the inlet to the ECO reactor.

#### Task 4: Determine the Effect of Wet Electrostatic Precipitator and Scrubber Operating Parameters on Mercury

Task 4 is described in the Cooperative Agreement as testing to determine the effect of scrubber and Wet Electrostatic Precipitator (WESP) operating parameters on the ability of each unit to capture and retain Hg<sup>2+</sup>. Parameters to be tested include scrubber liquid composition, flow rate and temperature and WESP operating voltages and temperature. As part of this task, Powerspan developed a method to quantify the mercury content of the scrubbing and WESP liquid streams.. The analysis method tested and used is described in Appendix A of this report.

The scrubbing system in the ECO process is segregated into two sections. The first section (or lower loop in the commercial design) saturates the flue gas exiting the barrier discharge reactor through evaporative cooling, concentrating the solution for removal of the byproduct. Liquid from this section is also used to capture ammonia vapor in the flue gas exiting the second scrubber section. The second scrubber section (or upper loop) captures SO<sub>2</sub> and NO<sub>2</sub> produced by the barrier discharge reactor. Ammonia is added to the second scrubber section to maintain scrubber pH. Water is added to both the first and second scrubber sections to maintain solution concentrations. Effluent from the second scrubber section is directed to the first scrubber section. The WESP is periodically washed with fresh water and the wash liquid is also directed to the first scrubber section. A portion of the liquid in the first scrubber section is continuously recirculated through an ash filter, manufactured by Global Filtration. Use of the filter is necessary due to the higher than expected ash loading on the process.

The concentrations of Hg and sulfate (SO<sub>4</sub><sup>2-</sup>) measured over 4 days of operation are presented in Figure 7. The figure shows concentrations in the first (Tank 1) and second (Tank 2) scrubbing sections. At the start of the test the scrubber liquid tanks were filled

with fresh water. It can be seen that the concentrations of Hg and  $\text{SO}_4^{2-}$  increase as operations proceed, with final concentrations dependant on the water addition rate, discharge reactor power, flue gas flow rate and gas moisture content. Higher concentrations of Hg and  $\text{SO}_4^{2-}$  are expected in Tank 1 than in Tank 2 due to the evaporation of water in the first scrubber section.

Measurement of solution concentrations such as those shown in Figure 7 are performed routinely at the ECO pilot and will be used in conjunction with measurements of the gas phase Hg concentration and particulate Hg content to perform material balances on the ECO system. Testing in Task 4 will include altering the water addition rates to the two scrubber sections to determine the influence of scrubber concentration on the ability to retain captured Hg.

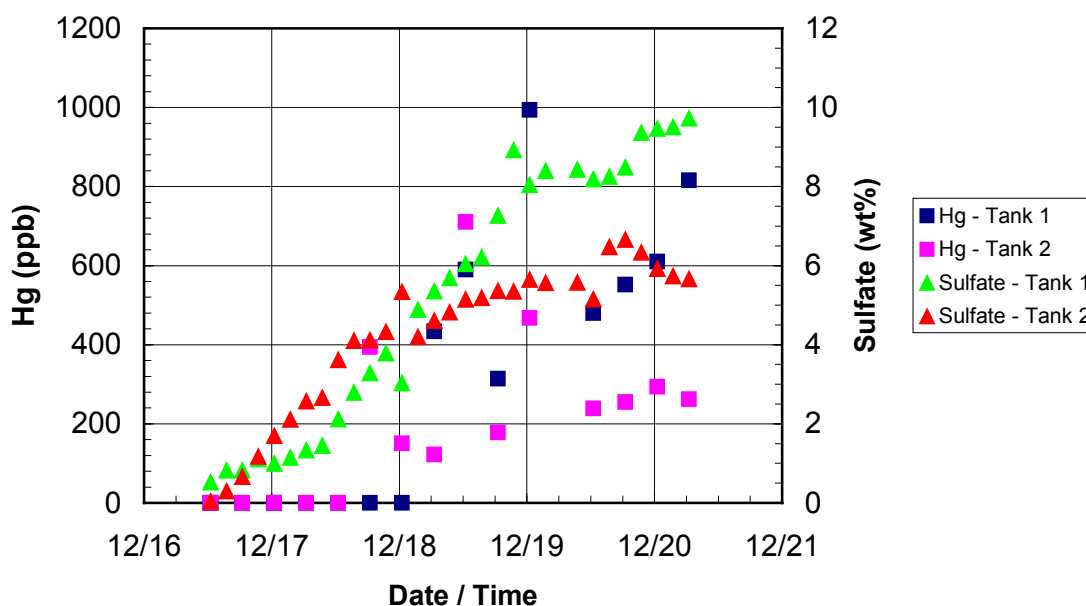


Figure 7 - Hg and Sulfate Concentrations in Scrubber Liquids

Although the absence of gas inlet Hg concentration prevents a material balance, the values recorded in the liquid are encouraging.

## Task 6: Determine Available Processes to Remove Mercury from Liquid Effluent Streams

Task 6 is meant to determine the best means for removing Hg from the effluent produced by the ECO process. Since the last quarterly report, testing in this area has focused on the following:

1. Processing effluent to remove mercury from the R.E. Burger pilot for crystallization studies.
2. Comparing the ability of Keylex and Mersorb LW<sup>®</sup> to remove mercury from ECO effluent streams.

Systems for treating synthetic and ECO Pilot effluent liquids have been constructed at the ECO pilot and in Powerspan's New Durham laboratories. The systems utilize Mersorb Mercury Adsorbent, a sulfur-impregnated activated carbon produced by NUCON International (Columbus, OH), to remove mercury in solution. They consist of two Mersorb beds in series and 0.5 micron filters on the inlet to and outlet of the beds for particle capture. The systems can process liquids at flowrates up to 0.5 gpm and have been used successfully to treat pilot liquids. Mercury levels in the effluent liquid have been reduced to less than the limit of detection from concentrations as high as 450 ppb. Subsequent crystallization of the effluent produced ammonium sulfate crystals contained 21% nitrogen and 24% sulfur, as would be expected for ammonium sulfate. Figure 8 shows three photographs of the crystals obtained during the crystallization process. Metals analysis of the crystals confirmed that the mercury concentration in the crystal product was below detectable limits.

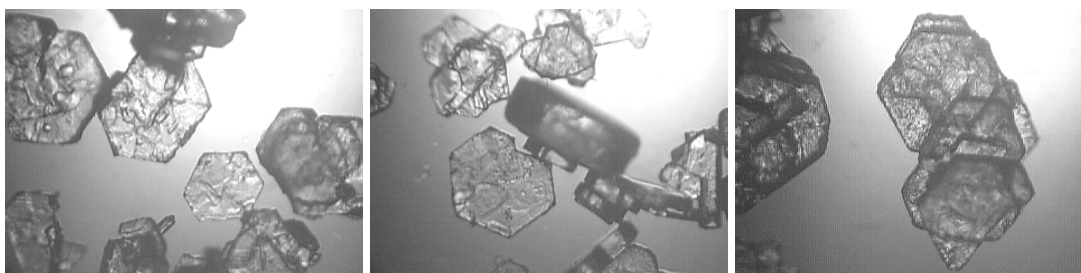


Figure 8 - Ammonium Sulfate Crystals

Another effort that is ongoing in the laboratory is comparing the ability of KeyleX to that of Mersorb for removing mercury from the effluent solutions.. KeyleX is a sulfonated chelating ion exchange resin produced by SolmeteX<sup>TM</sup> (Northborough, MA). It may be possible to reduce the size of the mercury removal system necessary for the ECO process by using KeyleX. Table 3 shows the performance as a function of pH for both KeyleX and Mersorb.

KeyleX			Mersorb LW		
pH	Initial Hg Concentration	Final Hg Concentration	pH	Initial Hg Concentration	Final Hg Concentration
	(ppb)	(ppb)		(ppb)	(ppb)
4	400	BDL	4	431	12
5	385	BDL	5	276	10
6	305	50	6	454	17

Table 3: Comparison of KeyleX and Mersorb for Mercury Removal from Effluent Solutions

The results in the table show KeyleX performs as well as Mersorb as a function of pH. Continued testing is necessary to determine the optimum flow rates and capacity of the KeyleX. Once this is done it will be possible to determine whether KeyleX or Mersorb are the best mercury removal materials for the effluent streams of the ECO Process.

#### Task 7: Technology Transfer

Task 7 is meant to provide for the transfer of information about the ECO process performance to the air pollution control community and to the public in general. During this quarter Powerspan personnel attended two events where information on the ECO process was presented. Details are presented below.

### *National Coal Council, Annual Fall Meeting*

On November 21, 2002, Chairman and CEO Frank Alix participated in a panel discussion on mercury emission controls at the National Coal Council's Fall meeting in Washington, D.C. The panel included executives from FirstEnergy, the National Energy Technology Laboratory, GE International, and ADA Technologies. The National Coal Council is a federal advisory committee to the Secretary of Energy.

### *Third Annual Power Generation Conference*

Frank Alix also delivered a presentation on October 17, 2002, at the Electric Power Generation Association's (EPGA) annual conference in Hershey, Pennsylvania. EPGA is a regional trade association of electric generating companies, including Allegheny Energy Supply, Exelon Generation, FirstEnergy Corp., Midwest Generation, PPL Generation, and Reliant Energy. Frank gave an update on ECO technology.

### Task Schedule

The following provides an estimate of the percentage of completion of each of the tasks defined in the Cooperative Agreement.

Task 1 - 90% Complete: Completion of this task is expected by the end of February 2003. Installation of batch sampling equipment and verification of measurement reliability are in progress. Once completed a Technical Report on the Task 1 results will be provided. The report will identify final configuration of the Hg CEMs systems at the pilot, along with the operating protocols and documentation verifying the adequacy of the protocols.

Task 2 - 60% Complete: Data with continuous addition of Hg to the flue gas stream, raising the inlet Hg concentration to 10 to 40  $\mu\text{g}/\text{Nm}^3$  is required.

Task 3 – Not started: Testing defined in this task requires reliable measurement of elemental and total Hg entering the barrier discharge reactor. Testing will begin at the end of February.



Task 4: 10% Complete: Testing has begun on scrubber and WESP parameters that influence the capture and retention of oxidized Hg.

Task 5 – Not started: Testing under this task requires completion of Tasks 3 and 4.

Task 6 - 75 % Complete: Testing continues at the pilot and in the laboratory. Pilot testing is focused on Hg removal from the liquid product stream. Laboratory testing on the capacity of adsorbents for Hg capture at liquid conditions representative of ECO liquids is ongoing.

Task 7 - 50 % Complete: The technology transfer task is ongoing throughout the course of the project.

Task 8 – Not started.

#### Financial Information Update

A brief table outlining the budget and expenditures to date is provided below.

Category	Expended Amounts (to date)	Budgeted Amounts (to date)
Personnel	\$ 629,914	\$ 718,815
Travel	\$ 118,965	\$ 82,520
Equipment	\$ 240,266	\$ 257,540
Supplies	\$ 287,468	\$ 101,250
Contracts	\$ 79,489	\$ 149,775
Indirect Charges	\$ 104,986	\$ 111,416
Total Costs	\$ 1,461,087	\$ 1,421,316

**Table 4: Budget Summary**

## Appendix A: Mercury Measurement in Ash and Process Fluids

Developing a method to measure mercury in both liquid solutions and in particulate was necessary to be able to close a mercury material balance for the ECO process. The particulate analysis was straight forward, and the Modified ASTM Method D 6414-01 was verified for our system. The ECO process fluid proved to cause problems for the EPA Method 254.1 mercury analysis. Therefore, it was necessary to develop a new digestion procedure that would not interfere with the mercury measurement by cold vapor atomic absorption. The following is a discussion of the verification of the methods used to measure both liquid phase and particulate mercury in the ECO process.

### Particulate Mercury Analysis (Mercury in Coal Fly Ash)

The method used to measure particulate mercury is the Modified ASTM Method D 6414-01, “Standard Test Method for Total Mercury in Coal and Coal Combustion Residues by Acid Extraction or Wet Oxidation/Cold Vapor Atomic Absorption”. To verify the method a Standard Reference Material (SRM) 1633b, “Constituent Elements in Coal Fly Ash,” was digested and analyzed.

Two samples of the SRM were digested and analyzed by cold vapor atomic absorption and compared to the certified value for mercury in the SRM. The results of the analysis are listed below in table 5.

Sample	Concentration	Percent Recovery
SRM Hg Concentration	141 ± 19 ppb	-----
SRM 1633b-1	161 ppb	114%
SRM 1633b-2	157 ppb	111%

Table 5 – Results of SRM Analysis of Particulate Hg

The results of the verification show a mean (95% Confidence) of  $159 \pm 25$  ppb. The method is a reliable way to measure the particulate mercury in the ECO process.

## Mercury Analysis in Process Fluids

The Modified EPA Method 245.1, “Determination of Mercury in Water by Cold Vapor Atomic Absorption Spectrometry”, has been modified to measure mercury in the ECO process fluids. The method has been modified due to interferences created by the digestion of ECO process fluids and the subsequent analysis by cold vapor atomic absorption. Rather than a mixture of acids, only concentrated nitric acid is used to digest ECO process fluids.

Testing was done to investigate the reliability and reproducibility of the method with the modified digestion to perform mercury analysis in liquids. Duplicate samples were prepared by adding 5 mL of the test solution to 20 mL of ultra-pure concentrated (69%) nitric acid (JT Baker Ultrex Grade) in a BOD bottle. To one of the samples a known quantity of mercury standard solution (0.1  $\mu\text{g Hg/mL}$ ) was added. The BOD bottle was covered with foil and digested for 2 hours in a hot water bath at 90 to 95 °C. After 2 hours, it was removed from the hot water bath, cooled to room temperature, and diluted to volume with deionized water. Five milliliters of stannous chloride were added to the sample solution and it was sparged for analysis. Analysis is done using a Buck Model 400 A Mercury Analyzer. A schematic of the analysis train is shown in figure 9.

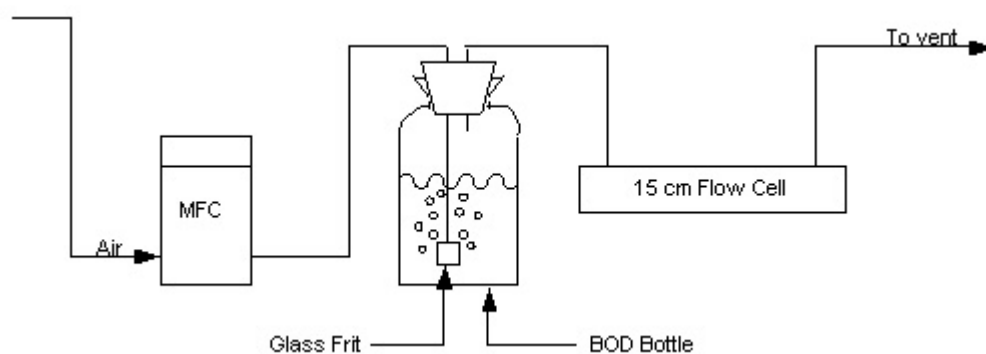


Figure 9 – Analysis Train for Mercury Analysis

Testing was performed both on synthetic solutions and on process fluid samples obtained at the R.E. Burger ECO pilot. For each test, duplicate solutions were analyzed; one was

analyzed unmodified and the second was spiked with a known quantity of mercury. The results of this testing is shown in table 6. The test shows that the digestion process developed for the ECO process fluids does not interfere with the measurement of mercury by cold vapor atomic absorption. The test solution compositions are proprietary, however solution 1 contains only the component from the ECO process fluid that required the modification of the EPA method 245.1 digestion. Solution 2 contains the ‘problem’ component as well as the other major components in the process fluids.

	<b>Expected Hg Concentration (µg)</b>	<b>Analyzed Hg Concentration (µg)</b>	<b>Percent Error</b>
Solution 1-1a	0.00	0.00	0
Solution 1-1b	0.40	0.27	-32.3
Solution 1-2a	0.00	0.00	N/A
Solution 1-2b	0.15	0.20	-22.8
Solution 2-1a	0.00	0.00	0
Solution 2-1b	0.40	0.46	14.1
Solution 2-2a	0.00	0.03	N/A
Solution 2-2b	0.40	0.47	16.9
Burger Plant Solution-1	N/A	0.21	N/A
Burger Plant Solution-2 (Spike)	N/A	0.375 (Spiked with 0.15 µg Hg) Delta = 0.375-0.211 = 0.164 µg	9.3

Table 6 – Results of Digestion Verification

It has been concluded from the data that the results are consistent with error values obtained in EPA Method 245.1. At a known concentration of 0.41 µg of mercury, the method reports a standard deviation of 0.112 µg of mercury, which corresponds to a range of 0.522 µg to 0.298 µg and an error of  $\pm 27.3\%$ . At a know concentration of 0.06 µg of mercury the method reports a standard deviation of 0.039 µg of mercury, which corresponds to a range of 0.099 to 0.021 µg and a resulting error of  $\pm 65\%$ .

### Instrument Precision Testing

Analysis on five identically prepared samples was done to test the precision of the Buck Analyzer. The samples were prepared by adding the same quantity of mercury standard to 20 mL of digestion solution in a BOD bottle. An aliquot of Solution 2 was then added to each of the bottles. The bottles were digested, diluted to volume and analyzed for mercury content. The results of the test are shown in table 7. The statistical analysis shows a standard deviation of 0.0006  $\mu\text{g Hg}$ , with an average value of 0.057  $\mu\text{g}$ . The percent relative standard deviation is 1.1 % and the mean (95% confidence) was  $0.575 \pm 0.007$ .

	Concentration ( $\mu\text{g Hg}$ )
Sample 1	0.566
Sample 2	0.580
Sample 3	0.573
Sample 4	0.580
Sample 5	0.575

Table 7 - Results from Instrument Precision Testing

The conclusion of this testing is that the Buck Analyzer is providing reliable and reproducible measurements

### Instrument Linearity Testing

The final verification done of the mercury measurements for the ECO process was a linearity check of the instrument. Seven solutions were prepared for analysis by adding known amounts of mercury to them. The solutions were digested by the method verified above and analyzed. The results are shown in table 8 and figure 10. The figure includes the 95% confidence intervals for the measurements.

Solution	Mercury Mass ( $\mu\text{g}$ )	Absorbance
1	0.04	0.009
2	0.06	0.015
3	0.08	0.022
4	0.15	0.038
5	0.25	0.063
6	0.40	0.102
7	0.60	0.149

Table 8 – Results from Instrument Linearity Check

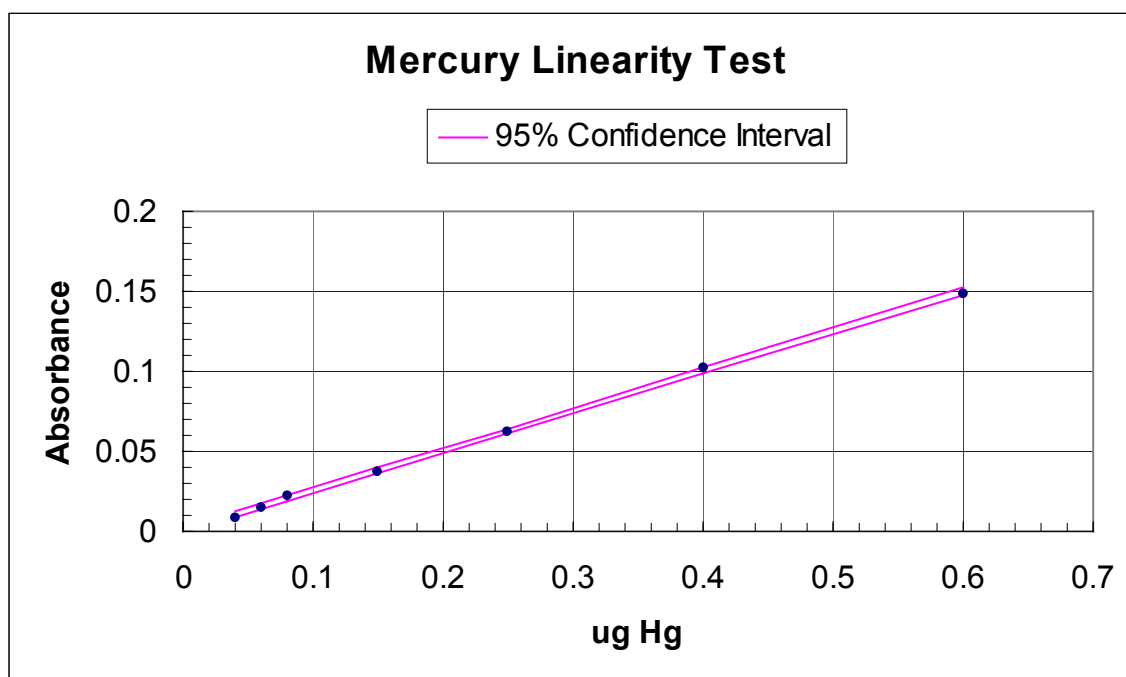


Figure 10 – Mercury Analyzer Linearity Check with 95% Confidence Intervals

#### Statistical Analysis

Correlation Coefficient: 0.9994

Y intercept: 0.0006

Slope: 0.2494

This test clearly shows the Buck Analyzer responds linearly from 0.04 to 0.6  $\mu\text{g}$  of mercury.

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